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PRODUCTION OF A BULK CONCENTRATE FROM A PORTUGUESE PYRITE ORE FOR SHERRITT GORDON MINES LIMITED

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by

A.I. Stemerowicz*

SUMMARY

At the request of Sherritt Gordon Mines Limited a Portuguese pyrite ore containing 3.00% zinc, 1.16% lead and 0.84% copper was treated in the Continuous Process Development Unit to produce a quantity of bulk concentrate for process development studies.

The bulk flotation method developed by Minimet Recherche in France was first tried in batch tests prior to the continuous run. At a grind of 79% minus 25 µm, 150.6 kg of bulk concentrate was produced assaying 20.85% zinc, 5.96% lead and 5.10% copper. Estimated recoveries in the bulk concentrate were 74.1% for zinc, 63.6% for lead and 68.9% for copper. Because the continuous cleaner flotation cells did not perform satisfactorily, it was necessary to resort to batch cleaning of the bulk rougher concentrate.

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INTRODUCTION

Request for Production of Bulk Concentrate

In September 1978 the Sherritt Research Centre of Sherritt Gordon Mines Limited, Fort Saskatchewan, Alberta approached CANMET about the possibility of using CANMET facilities to produce a bulk zinc-copper-lead concentrate from a Portuguese pyrite ore. They had an agreement with their Portuguese client to carry out a process development study to recover zinc, copper and lead from the bulk concentrate. About 300 kg was required for the study and was to be produced from 3000 kg of ore.

Because of the relatively small quantity of bulk concentrate required it was only feasible to carry out this work in a mini pilot plant such as the CANMET 50 kg/h Continuous Process Development Unit (CPDU) which, as far as it is known, is the only facility of its kind in North America.

An official request for the work was made by Dr. H. Veltman, Director of the Sherritt Research Centre, in his letter of December 12, 1978 and approved by the Director-General, CANMET on January 4, 1979. The work was to be carried out on a cost recovery basis with the costs estimated at $14,691.

Bulk Flotation Procedure

The procedure to be used in the CPDU for the production of the bulk concentrate was that develop d on a bench-scale by Minimet Recherche in France. It was agreed to have one of the

flotation engineers of Sherritt Gordon's client present to observe and assist in the operation of the CPDU. Before proceeding with continuous treatment in the CPDU, it was also agreed that some preliminary batch testing should be performed so that CANMET personnel could become familiar with the test procedure and response of the ore to flotation.

Mineralogy and Composition of the Ore

The ore can be characterized as a massive, very fine-grained sulphide ore and is similar to the New Brunswick ores. The ore deposit is composed of two main ore types — "Moinho" (médio and rico) and "Feitais" whose composition is given in Table 1. Sphalerite, galena and chalcopyrite are the most important economic minerals and occur in a matrix of pyrite. Of lesser importance are the sulphosalts, a variety of which are found in both ore types. In the Feitais ore 35% of the copper is accounted for by tetrahedrite, while in the Moinho médio,10% of the lead content is attributed to bournonite. The average iron content of the sphalerite in the Moinho and Feitais ores has been determined to be 5% and 2.5%, respectively. It has been estimated that the average grain size of the sphalerite and chalcopyrite is in the 20-25 μm range while that of galena and lead sulphosalts is about 10 μm.

TABLE 1

Composition of Portuguese Ores *

Constituent	Moinho medio ore	Feitais ore
Cu	1.20%	0.56 %
Zn	3.0 %	3.0 %
Pb	1.18%	1.3 %
S	48.1 %	46.4 %
Fe	41.2 %	39.2 %
Ag	34 ppm	27 ppm
Au	0.9 ppm	0.8 ppm
Hg	76 ppm	94 ppm
As	0.73%	0.56 %
Sb	0.07%	0.105%
Co	214 ppm	55 ppm
Bi	185 ppm	100 ppm
Cd	55 ppm	57 ppm
Se	40 ppm	28 ppm
Te	<50 ppm	<50 ppm
Sn	<50 ppm	<50 ppm
Ni	32 ppm	27 ppm
SiO_2	3.4 %	6.92 %

* Analysis by Portuguese

Projected Grades and Recoveries for Bulk Flotation

The assay for a typical bulk concentrate was indicated* follows: 21.88% zinc, 4.34% lead, 6.10% copper and 21.92% iron. Such a concentrate had been produced from feed which contained 2.50% zinc, 1.03% lead and 0.79% copper. Minimum recoveries expected for zinc, lead and copper are 85%, 70% and 80%, respectively.

* Information supplied by Sherritt Gordon's Portuguese client

Ore Shipment

A shipment of ore consisting of 10 steel drums, net weight about 3800 kg, was received on November 21, 1978. The drums had their covers welded on and contained ore crushed to -5 cm and stated to be a blend of 60% Moinho médio ore and 40% Feitais ore.

Crushing, Sampling and Analysis

The shipment of ore was crushed to -12 mm and then passed through an automatic sampling system to obtain a representative head sample of about 200 kg. In the process the ore from the various drums was well mixed. The head sample was split into quarters and one of the quarters was further crushed to -1.65 mm (10 mesh) and riffled into 2000-g lots. One of these 2000-g lots was chosen at random for analysis while the remaining lots were used in the batch tests.

For the continuous run in the CPDU the remainder of the ore was crushed to -2.35 mm (8 mesh).

TABLE 2

Analysis of Head Sample

Constituent	%
Zn	3.00
Pb	1.16
Cu	0.84
Fe	41.4
S	48.1
Insolubles	4.08

BATCH TESTS

Bulk Flotation Using Minimet Procedure and Modifications

The Minimet bulk flotation procedure, details of which are given in Table 3, differs from conventional practice in two respects:

1. The use of a very high pH (12+) in both roughers and cleaners — normally the pH is kept below 10 to minimize galena depression.
2. The addition of all of the collector at the start of flotation rather than stage-feeding it during flotation.

In the Minimet procedure a grind of 80% -33 to 35 µm was specified. This was tried but found to be inadequate. The desired recovery required a finer grind to 80% - 29 µm. In one test a still finer grind of 80% -23 µm was employed. These grinds were obtained by grinding a 2000-g test charge of -10 mesh ore for 45, 60 and 90 minutes in a 7 x 14 Denver Laboratory rod mill. Size analyses are given in Table 4.

Table 5 compares the results obtained using the Minimet procedure at various grinds and with modifications in procedure as noted. The purpose of these modifications was to determine whether the procedure could be simplified without affecting results.

TABLE 3

Minimet Bulk Flotation Procedure

Operation	Unit Used	Time (min)	% Solids	pH	Reagent additions, g/t				
					NaCN	Lime	$CuSO_4$	Z-6[5]	MIBC[6]
Grinding	7 x 14 RM[1]	60	65		100				
Conditioning-1	pail[2]	3	35	12.4		3000			
Conditioning-2	pail	3		11.9			600		
Conditioning-3	4-in aerator	7		11.7					
Bulk rougher flotation	1000-g cell	7½	35	12.2		500		100	10
Bulk ro conc[3] regrinding	8-in.[4] BM	30	50						
Bulk cleaner-1	500-g cell								
Conditioning-1		2		12.3	20	600			
Conditioning-2		2					200		
Float		7	25						10
Bulk cleaner-2	250-g cell	4	25	12.0					

Notes: (1) Rod charge - 20 kg, ranging in diameter from 10 to 32 mm
(2) Agitated with flotation mechanism
(3) Bulk rougher concentrate filtered and cake washed into ball mill with 750 cc filtrate
(4) Ceramic ball mill charged with 12 mm diameter ceramic balls
(5) Potassium amyl xanthate
(6) Methyl isobutyl carbinol (frother)

TABLE 4

Size Analysis of Batch Rod Mill Grinds

Size Fraction	Diameter (μm)	Wt % retained		
		45 min grind	60 min grind	90 min grind
400 mesh	38	18.7	5.4	2.8
Conc 1 o'flow*	24.4	21.3	20.9	9.6
Conc 2 o'flow	18.6	17.4	21.3	21.6
Conc 3 o'flow	13.5	10.4	13.1	17.1
Conc 4 o'flow	9.3	9.3	11.5	15.0
Conc 5 o'flow	7.2	4.2	5.1	6.7
Conc 5 u'flow	<7.2	18.7	22.7	27.2
Total		100.0	100.0	100.0
K_{80} μm		35	29	23

* Warman cyclosizer fractions

The best results were achieved using the Minimet procedure as given at a grind of 80% -29 μm (Test 8) but the lowest losses of zinc and copper in the tailing were obtained for the test in which a fine grind of 80% -23 μm was employed (Test 5). Note that unlike the zinc and copper, the lead loss in the tailing did not vary appreciably with the grind (compare Test 5 with Test 10).

TABLE 5

Summary of Batch Results Using Minimet Procedure and Various Modifications

Test No.	Grind K_{80}, µm	Remarks	Product	Wt %	Analysis %			Distribution %		
					Zn	Pb	Cu	Zn	Pb	Cu
5	23	Modified Minimet Procedure (1)	Cl conc	11.56	22.80	4.33	5.90	87.7	44.7	77.7
			Cl tail	17.96	1.38	2.04	0.62	8.3	32.7	12.7
			Ro tail	70.48	0.17	0.36	0.12	4.0	22.6	9.6
			Feed	100.00	3.00	1.12	0.88	100.0	100.0	100.0
7	29	Minimet Procedure as given	Cl conc	12.06	22.20	5.23	5.75	90.2	56.5	78.9
			Cl tail	17.95	0.81	1.33	0.49	4.9	21.5	10.0
			Ro tail	69.99	0.21	0.35	0.14	4.9	22.0	11.1
			Feed	100.00	2.97	1.12	0.88	100.0	100.0	100.0
8	29	Modified Minimet Procedure (2)	Cl conc	11.33	23.70	4.20	5.68	90.0	43.1	72.2
			Cl tail	17.79	0.72	1.98	0.72	4.3	31.9	14.3
			Ro tail	70.88	0.24	0.39	0.17	5.7	25.0	13.5
			Feed	100.00	2.98	1.11	0.89	100.0	100.0	100.0
10	35	Minimet Procedure as given	Cl conc	9.96	25.30	4.80	5.05	84.9	44.5	61.6
			Cl tail	21.50	1.10	1.53	0.63	8.0	30.6	16.6
			Ro tail	68.54	0.31	0.39	0.26	7.1	24.9	21.8
			Feed	100.00	2.97	1.07	0.82	100.0	100.0	100.0
11	29	Modified Minimet Procedure (3)	Cl conc	10.50	24.00	4.15	4.73	83.0	39.1	58.7
			Cl tail	19.43	1.71	1.94	1.04	11.0	33.9	23.9
			Ro tail	70.07	0.26	0.43	0.21	6.0	27.0	17.4
			Feed	100.00	3.03	1.11	0.85	100.0	100.0	100.0

(1) Bulk rougher concentrate not reground before cleaning
(2) Used only one conditioning stage for lime and $CuSO_4$, also added NaCN to regrind mill instead of after grinding
(3) Used only one conditioning stage for lime and $CuSO_4$, rougher concentrate not reground before cleaning

Regrinding of the bulk rougher concentrate before cleaning resulted in higher recoveries in the bulk cleaner concentrate (compare Test 11 with Test 7).

Bulk Concentrate Production by Combining Selectively-Flotated Lead and Zinc Concentrates

This scheme for bulk concentrate production was effective on New Brunswick ore and it was deemed worthwhile to try it on the Portuguese ore. The procedure is given in Table 6 followed by the results in Table 7.

The selective flotation scheme gave considerably higher lead recovery in the bulk concentrate than did any of the bulk flotation tests. Zinc and copper recoveries were on a par with those obtained for bulk flotation. Note the much lower losses of zinc, lead and copper in the selective flotation cleaner tailings when compared to the bulk flotation cleaner tailings. This accounts for the high recoveries obtained when the lead and zinc concentrates were combined to form the bulk concentrate.

CONTINUOUS TESTS

Flowsheet

The flowsheet and the reagents and conditions used for the production of the bulk concentrate are given in Figure 1 and Table 8. Note that open-circuit grinding is employed because classification methods for fine grinds have as yet not been developed for the CPDU. As indicated, the bulk rougher

TABLE 6

CANMET Selective Flotation Procedure for Bulk Concentrate Production

Operation	Unit used	Time (min)	% Solids	pH	Reagent additions, g/t				
					Na_2CO_3	NaCN	AF242[1]	Z-6	DF250[2]
Grinding	7 x 14 RM	60	65	9.8	2500	200	25		
Conditioning	Aerator	10	35					10	
Lead rougher	100-g cell		35						
Stage 1		1/2		9.0					
Stage 2		1/2						10	
Stage 3		1					12		10
Lead Cl-1	250-g cell	2		8.8					
Lead Cl-2	250-g cell	2		8.7					5
Lead Cl-3	250-g cell	2		8.5					5
Filtered lead Cl tails & added to zinc conditioning									
					$CuSO_4$	Lime	Z-200[3]	Z-6	DF250
Zn conditioning	1000-g cell	10		11.7	600	2000			
Zn rougher	1000-g cell								
Stage 1		1					20	25	
Stage 2		1					20		
Stage 3		4					10		10
Zn ro conc regrind	8-in BM	30	50						
Conditioning	500-g cell	3		11.1	200	250			
Zn cleaner-1	500-g cell	6						20	30[4]
Zn cleaner-2	250-g cell	4		11.2		50			10
Zn cleaner-3	250-g cell	3		10.7					10

Notes: (1) Aerofloat 242 Promoter - dithiophosphate collector
(2) Dowfroth 250 - polypropylene glycol frother
(3) Dow Chemical Co. selective zinc collector - composition unknown
(4) Stage fed

TABLE 7

Batch Results for Bulk Concentrate Production by Selective Flotation (Test 12)

Product	Wt %	Analysis %			Distribution %		
		Zn	Pb	Cu	Zn	Pb	Cu
Lead conc	5.54	4.40	12.10	10.70	8.3	59.4	69.4
Zinc conc	7.32	33.20	1.42	1.20	83.0	9.2	10.3
Zinc cleaner tail-3	0.28	1.59	1.10	0.64	0.2	0.3	0.2
Zinc cleaner tail-2	0.89	0.68	0.93	0.41	0.2	0.7	0.4
Zinc cleaner tail-1	5.62	0.48	0.67	0.27	0.9	3.3	1.8
Zinc rougher tail	80.35	0.27	0.38	0.19	7.4	27.1	17.9
Feed (calcd)	100.00	2.93	1.13	0.85	100.0	100.0	100.0
Lead + Zinc conc = Bulk conc	12.86	20.79	6.02	5.29	91.3	68.6	79.7

TABLE 8

CPDU Reagents and Conditions for Production of Bulk Concentrate

Operation	Retention time, (min)	% Solids	pH	Reagent additions, g/t				
				NaCN	Lime	$CuSO_4$	Z-6	DF250
Grinding - stage 1	30	65		100				
Grinding - stage 2	70	60						
Conditioning	25	33	12.2		4400	1000		
Aeration	27	33						
Bulk ro flotation	48	33						
Cell 1			12.5		880		120	
Cell 2							35	25
Bulk cleaners								
Stage 1		30	11.9					*
Stage 2		25	11.7					*
Stage 3		20	ca 11.5					*

* as required

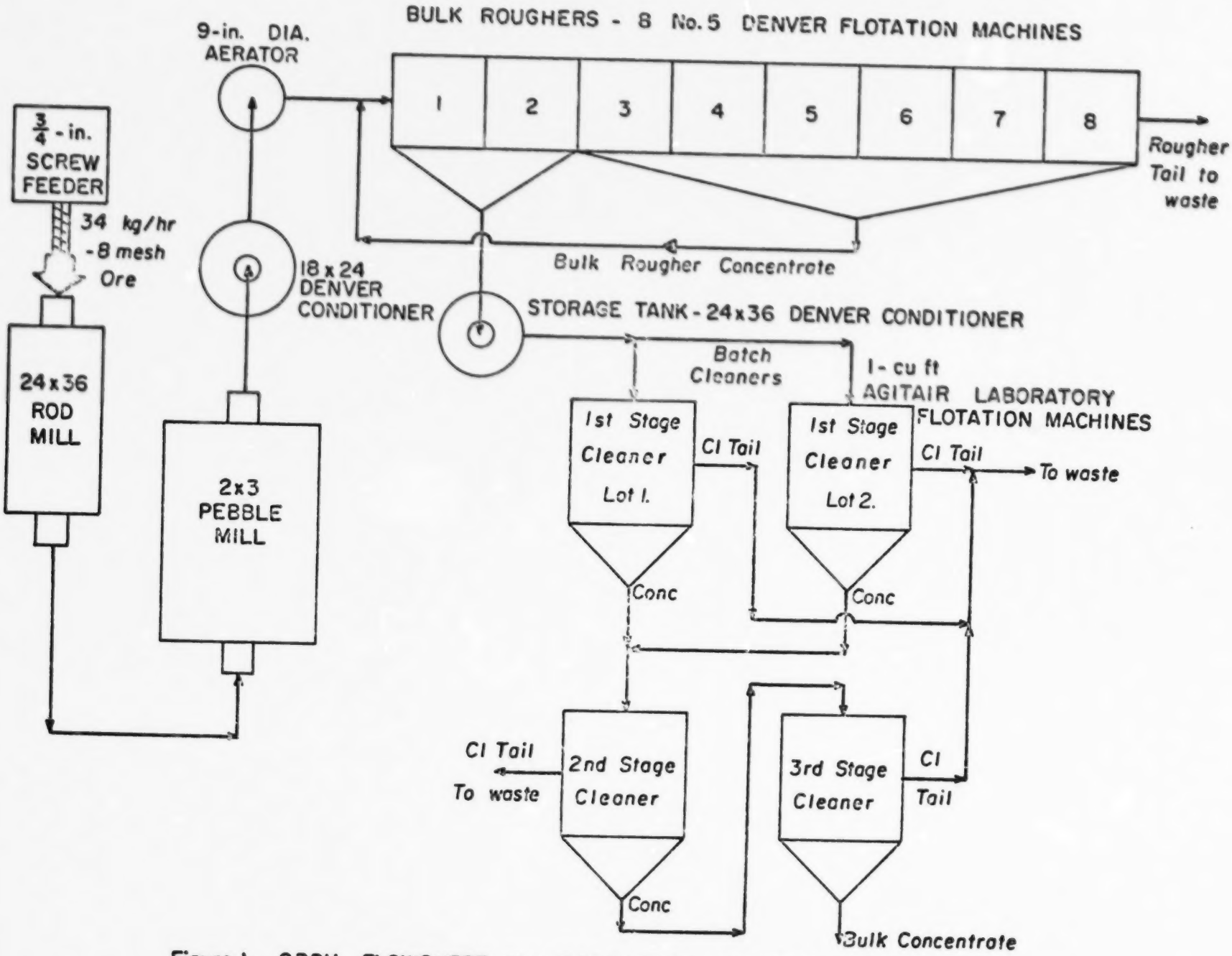

Figure 1. CPDU FLOWSHEET FOR PRODUCTION OF BULK CONCENTRATE

concentrate produced in the CPDU was batch cleaned using a 1 cu ft Agitair batch flotation machine. Continuous cleaning in the 2.5-litre CPDU cleaner flotation machines was tried initially but these machines, which are still under development, have not been consistent in giving satisfactory cleaning efficiency.

Metallurgical Problems

A severe and unexpected recovery problem was encountered at the start of operations and was not fully solved until Run No. 8. By carrying out batch tests on ground samples taken at various points in the CPDU and comparing results obtained with CPDU results, it was established that the following factors contributed to the low recoveries obtained in the initial test runs:

1) Conditioning, 1st stage - the original conditioner gave poor mixing because of a badly-worn impeller.
2) Conditioning, 2nd stage (aerator) - because the discharge outlet of the aerator was located below the bottom of the discharge baffle there was a possibility that some of the pulp was not being fully aerated because of short-circuiting.
3) Grinding - contact time in the ball mill used for the second-stage grind was about 2 hours. This long contact time with steel appeared to be detrimental to flotation.

4) Rougher Flotation - the 0.3-cu ft Agitair cells used for roughing did not perform satisfactorily - too much turbulence at the surface of the pulp appeared to interfere with froth formation.

After rectifying all of the above problems either by equipment replacement or modification, no further difficulties were encountered in reproducing batch results in the CPDU.

Results

Table 9 compares CPDU results with those of a batch test at a similar grind. Included in the comparison are the results of some of the initial runs in which low recoveries were obtained. In Run No. 2 and No. 4 the bulk rougher concentrate was cleaned twice in 2.5-litre CANMET flotation machines. The cleaners were cut out of the CPDU circuit starting with Run No. 6 and replaced by batch cleaning.

From the comparison it can be seen that there was a progressive improvement in recovery as the various equipment changes were made. Finally, rougher flotation recoveries in the CPDU equalled those obtained in batch tests.

One set of CPDU products was submitted for silver assays which are given in Table 10.

Silver recovery in the bulk concentrate was very low when compared to the lead recovery. Generally, for most ores the two are related and follow each other closely.

TABLE 9

Comparison of CPDU and Batch Results

Test No.	Grind % passing 25 µm	Product	Analysis, %			Distribution, %		
			Zn	Pb	Cu	Zn	Pb	Cu
CPDU 2 (1)	80.6	Bulk Cl conc	15.34	5.54	8.02	26.4	23.8	48.0
		Bulk ro tail	2.37	0.98	0.48	73.6	76.2	52.0
		Feed (calcd)	3.05	1.22	0.87	100.0	100.0	100.0
		Bulk ro conc	9.81	3.06	4.32			
CPDU 4 (2)	83.2	Bulk Cl conc	15.14	3.30	4.58	62.9	37.0	65.6
		Bulk ro tail	1.30	0.82	0.35	37.1	63.0	34.4
		Feed (calcd)	3.06	1.14	0.89	100.0	100.0	100.0
		Bulk ro conc	9.68	2.44	2.94			
CPDU 6 (3)	78.4	Bulk ro conc	7.97	2.51	2.20	89.8	79.4	83.9
		Bulk ro tail	0.47	0.34	0.22	10.2	20.6	16.1
		Feed (calcd)	3.04	1.08	0.90	100.0	100.0	100.0
CPDU 8 (4)	78.3	Bulk ro conc	10.95	3.39	3.01	93.3	78.4	84.4
		Bulk ro tail	0.27	0.32	0.19	6.7	21.6	15.6
		Feed (calcd)	2.99	1.10	0.91	100.0	100.0	100.0
Batch 14 (5)	81.6	Bulk ro conc	9.34	2.72	2.62	94.3	75.0	85.9
		Bulk ro tail	0.25	0.40	0.19	5.7	25.0	15.1
		Feed (calcd)	3.03	1.11	0.93	100.0	100.0	100.0

Notes: (1) Results with initial CPDU equipment in operation
(2) Results after first stage conditioner was replaced and discharge point of aerator was taken off at a higher location behind discharge baffle
(3) Steel ball mill with a steel ball charge was replaced with a ceramic-lined mill with pebble charge - this resulted in a reduction in retention time in the mill from about 2 hours to about 1 hour
(4) 0.3 cu ft Agitair flotation machines replaced with 0.25 cu ft Denver No. 5 flotation machines
(5) Test on sample from CPDU screw feeder

TABLE 10

Metallurgical Balance for Silver, CPDU Run No. 8

Product	Wt %	Ag assay, g/t	Ag distribution, %
Bulk ro conc	25.47	73.0	48.0
Bulk ro tail	74.53	27.1	52.0
Feed (calcd)	100.00	38.7	100.0
Feed (assay)		39.1	

Batch Cleaning

Originally, the intention was to produce a bulk concentrate with a target grade of 22% as specified. However, because a substantial portion of the ore had been treated in the initial runs without producing a corresponding amount of bulk concentrate of the desired grade, the target grade for batch cleaning was lowered to 20% zinc to ensure that an adequate amount was produced from the remaining ore.

The CPDU bulk rougher concentrate was first accumulated in a 24 x 36 Denver conditioner and then pumped to the 1-cu ft Agitair batch cell for cleaning. Generally, three stages of cleaning were required to upgrade the concentrate to the desired grade.

In Table 11 the results of batch cleaning of CPDU rougher concentrate are given and compared with continuous cleaning in CPDU Run No. 4. Also included in the comparison are the results of a batch test done on a sample of ore from the CPDU screw feeder.

TABLE 11

Comparison of Cleaning Results

Test and remarks	Product	Wt %	Analysis, %			Distribution, %		
			Zn	Pb	Cu	Zn	Pb	Cu
Batch cleaner No. 15 and 16 on CPDU bulk rougher conc. -2 stages	Cl conc	50.59	17.85	5.61	5.06	84.5	84.7	87.7
	Cl tail (calcd)	49.41	3.36	1.04	0.73	15.5	15.3	12.3
	Feed (assay)	100.00	10.69	3.35	2.92	100.0	100.0	100.0
Batch cleaner No. 27 on CPDU bulk rougher conc -3 stages	Cl conc	40.33	22.63	6.93	6.10	74.2	77.6	75.5
	Cl tail (calcd)	59.67	5.32	1.35	1.34	25.8	22.4	24.5
	Feed (assay)	100.00	12.30	3.60	3.26	100.0	100.0	100.0
Continuous cleaning, CPDU Run No. 4 - 2 stages	Cl conc	37.95	15.14	3.30	4.58	59.4	55.9	65.4
	Cl tail	62.05	6.34	1.59	1.48	40.6	44.1	34.6
	Feed (calcd)	100.00	9.68	2.24	2.66	100.0	100.0	100.0
Batch Test No. 15 on CPDU rod mill feed - 3 stages	Cl conc	54.77	22.70	4.30	5.13	92.7	67.9	80.1
	Cl tail	45.23	2.16	2.46	1.53	7.3	32.1	19.8
	Feed (assay)	100.00	13.41	3.47	3.50	100.0	100.0	100.0

Batch cleaning in the large 1-cu ft Agitair cell was much more efficient than continuous cleaning in the 2.5 litre CPDU flotation cells. However, when compared to the batch cleaning results obtained using the smaller 1 and 2-litre Denver laboratory cells, the zinc recovery in the cleaner concentrate was much lower although the lead recovery was higher (compare batch cleaner No. 27 with batch test No. 15). No explanation can be offered for the divergence in lead and zinc recoveries.

Average grade and weight of bulk concentrates produced by batch cleaning are given in Table 12 along with estimated recovery.

TABLE 12

Bulk Concentrates Produced by Batch Cleaning

Dry wt kg	Analysis, %			Recovery, %		
	Zn	Pb	Cu	Zn	Pb	Cu
150.6	20.85	5.96	5.10	74.1	63.6	68.9

Some lower-grade bulk concentrates produced at the beginning of the continuous run were also shipped at the request of Sherritt Gordon. This material amount to 52.1 kg and assayed 13.05% zinc, 3.82% lead and 4.80% copper.

CONCLUSIONS

Because of the poor performance of the 2.5-litre CPDU cleaner cells it was not possible to produce bulk concentrate on a continuous basis with the projected minimum recoveries of 85% for zinc, 70% for lead and 80% for copper. It is believed that the poor performance is related to the high froth surface area to volume ratio of these small cells.

When the expedient of batch cleaning the bulk rougher concentrate in a 1-cu ft Agitair cell was adopted, it was anticipated that results would be similar to those obtained in batch tests using the small Denver 500-g (2 litre) and 250-g (1 litre) laboratory cells. On this basis a zinc recovery of over 85% could be predicted but this was not the case. For an equivalent concentrate grade about 18% less zinc was recovered from the bulk rougher concentrate using the Agitair. However, lead recovery was about 10% higher.

In retrospect it might have been advantageous to grind the rougher concentrate and recondition with copper sulphate before cleaning. In batch tests this treatment resulted in higher recoveries in the cleaning step. Also, the author was at a disadvantage because he was not supplied with detailed metallurgical balances of Minimet batch tests. This data could have been helpful in diagnosing and rectifying some of the problems encountered.

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